ENGELHARD'S Q-5 REAGENT

DESCRIPTION

Engelhard's Q-5 Reagent is a dark gray, irregular shaped granule (8-14 mesh). The material is actually a highly active metal-containing, inert supported reagent. Engelhard's Q-5 retains oxygen by chemical reaction with the metal to form the metal oxide. The regeneration procedure is essentially a reduction of the metal oxide back to the metal. Reduced Q-5 has a light red cast which gradually, but not dramatically, runs dark gray upon oxidation. The reagent, as received, is in the oxidized state and must be reduced before first use.

It's no longer necessary to tolerate those (last few) ppms of oxygen in your laboratory gases. Ninety-nine percent of the oxygen present in inert and certain other gases can be eliminated by passage of the gas through a single tube or bed of Engelhard's Q-5 Reagent. And at ambient temperature too. No bothersome heating and cooling of the gas are necessary. Of course, if you prefer to treat the gas at a different temperature, Q-5 can do so – from 40°C to 200°C. When the Q-5 becomes saturated with oxygen, full activity is restored by a simple regeneration. Under normal use, the Q-5 will remain active through numerous cycles.

Engelhard's Q-5 Reagent has been used for several years in purifiers of laboratory glove boxes to maintain very low oxygen content in inert atmospheres. Certain reactive chemicals and fuels, high purity metals, radioactive materials, some biological species and many other items are very sensitive in oxygen. Treatment with Q-5 of the gases used for blanketing these materials eliminates oxygen problems. For handling those materials that are sensitive to both moisture and oxygen, Q-5 is often used in conjunction with molecular sieves* (water removal). This combination is operationally advantageous since both Q-5 and molecular sieves are operated and regenerated simultaneously under the same conditions.

The performance of various analytical instruments is adversely affected by trace oxygen contamination of carrier gases. For many analyses by vapor phase chromatography, the detector is sensitive to the trace oxygen and precise results are impossible. A simple tube of Q-5 in the carrier gas line does not complicate operations and prevents oxygen interference. This gas treatment with Q-5 is also beneficial to the operation of several other types of instruments.

***Union Carbide Corporation**
Engelhard's Q-5 Reagent is also used to purify the total inert gas supply of large multi-purpose laboratories. A central purification system, using Q-5 reactant, can be used to assure oxygen-free nitrogen for all needs in analytical, reaction inert blanketing, inert chambers, etc. Occasionally, purified nitrogen users will experience unexpected improvements in their experiments even when oxygen sensitivity was not expected.

Q-5 Reagent offers a simple, efficient means to rid oxygen from your gas system. It can be used for significant lengths of time with little attention and offers real insurance that oxygen interference will not occur.

DESIGN OF ENGELHARD'S Q-5 UNITS

The first question that must be answered in setting up a bed for Q-5 oxygen removal is: How much of Engelhard's Q-5 Reagent is needed for this job? The quantity depends on the desired maximum oxygen content of the effluent gas, the length of the regeneration interval desired and the pressure drop across the Q-5 which can be tolerated. The following terms are useful in this determination:

> **Efficiency: The percent of oxygen that is removed by Q-5.**
>
> **Dynamic Capacity: The quantity of oxygen retained by the Q-5, cc oxygen (STP)/g Q-5.**

As shown in Figure 1, the oxygen removal efficiency is a function of the Q-5 capacity, i.e., as the oxygen retention of the Q-5 increases, the efficiency decreases. One outstanding feature of the Q-5 performance is the high efficiency maintained up to significant capacity. The minimum oxygen removal efficiency for a given system will be set by the maximum oxygen level tolerable. For some applications where absolute minimum oxygen level must be maintained, a minimum efficiency of 95 to 98 percent may be chosen. For other applications, perhaps the removal of only 80-90 percent is necessary. In any case, the Q-5 will allow 98+ percent oxygen removal through the major portion of the cycle and will decrease to the design minimum during the last part of the cycle. At that time, the Q-5 is regenerated and full efficiency is restored.

In sizing a Q-5 unit, the first step is choosing the oxygen removal efficiency needed. Using this efficiency, the maximum capacity is

determined from Curve 1, Figure 1. The on-stream line (between regenerations) is calculated by the following formula:

$$\textbf{Time, Hour} = \frac{(\text{capacity, cc / g})}{(\text{space velocity, V / hr / V})(\text{initial concentration})}$$

Space Velocity = 3000 V/hr/V (Curve 1)

Table 1 also gives cycle lengths at various inlet oxygen levels. If the regeneration interval given by this calculation is not long enough for your practical purposes, it can be increased by using lower space velocities, i.e., more Q-5 for the same flow rate of gas. This space velocity is determined by linear proportions of the cycle time calculated previously to the cycle time desired, i.e.,

$$\textbf{Space velocity} = 3000 \times \frac{(\text{Calculated Cycle Time})}{\text{Desired Cycle Time}}$$

Note that space velocity is the volume of total gas per hour per volume of Q-5. For example, for a gas flow rate of 30,000 cc per hour and a Q-5 volume of 10 cc, the space velocity is

$$\frac{30{,}000\,cc / hr}{10\,cc} = 3000 \text{ cc/hr/cc}$$

For a given gas flow rate, as the catalyst volume used increases, the space velocity decreases.

If the cycle time for 3000 V/hr/V space velocity is longer than necessary, the Q-5 volume required can be minimized by using a higher space velocity. Of course, higher space velocity will give a somewhat lower capacity for a given efficiency, thus a new calculation, not just a simple proportioning, is necessary. A space velocity in excess of 6000 V/hr/V is not recommended.

Q-5 Requirements – Example:

Basis: **1000 cc/min inert gas**
20 ppm (vol.) oxygen
1 ppm (vol.) maximum oxygen desired

$$\textbf{Minimum efficiency} = \frac{20-1}{20} \times 100 = 95\%$$

From Figure 1, Curve 1, at 95 percent efficiency the capacity is 1.25 cc oxygen/cc Q-5. Using Formula 1,

$$\textbf{Cycle Time} = \frac{(1.25 \text{ cc / cc})}{(3000 \text{ cc / hr / cc})(20 \text{ x } 10^{-6})}$$

$$= \textbf{20.8 hr}$$

If a longer cycle time is desired, a lower space velocity should be used. For a cycle time of 100 hours:

$$\textbf{Space Velocity} = \frac{3000\,x\,20.8}{100} = \textbf{625 cc/hr/cc}$$

The Q-5 requirement would thus be:

$$\textbf{Q-5 Quantity} = \frac{(1000 \text{ cc / min x 60 min / hr})}{625 \text{ cc / hr / cc}}$$

$$= \textbf{96 cc Q-5}$$

OR

96 cc x 1 g/cc = 96 g Q-5

If the total pressure drop across a Q-5 unit exceeds the desired maximum, certain allowances can be made. Lower space velocities and decreased container length to diameter ratios will give lower pressure drop. A minimum length/diameter ratio of 1.0 is suggested.

TABLE 1

Q-5 CYCLE TIMES

3000 V/Hr/V

Oxygen Concentration ppm (vol.)	Efficiency %	Cycle Time, Hours 98	95	90	70
5		50	83	116	150
10		25	42	58	75
20		12.5	21	29	38
50		5.0	8.3	12	15
100		2.5	4.2	5.8	7.5
500		0.5	0.8	1.2	1.5

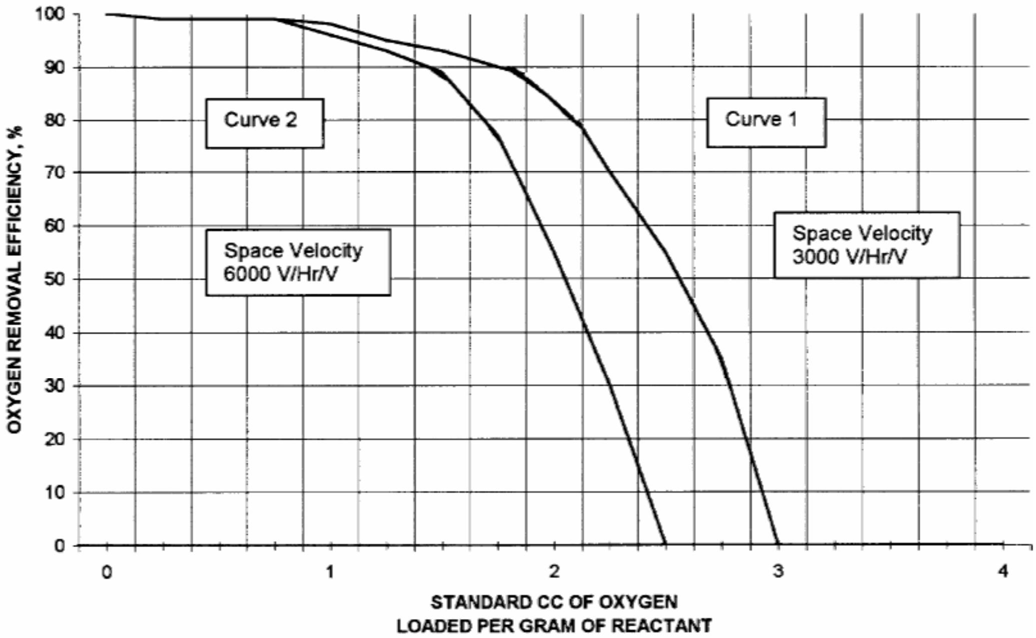
TABLE 2

Q-5 CYCLE TIMES

6000 V/Hr/V

Oxygen Concentration ppm (vol.)	Efficiency %	Cycle Time, Hours			
		98	95	90	70
5		16	35	47	60
10		8.0	17.5	23	30
20		4.0	8.8	12	15
50		1.6	3.5	4.7	6
100		0.8	1.8	2.3	3
500		0.16	0.35	0.47	0.60

FIGURE 1
DYNAMIC OXYGEN CAPACITY OF REACTANT
(AMBIENT TEMP)



ACTIVATION OF Q-5 REAGENT

Engelhard Q-5 Reagent, as purchased, is supplied in the oxidized state and must be reduced before first use. After reduction, the Q-5 should not be exposed to any source of oxygen or air. Thus, the reduction should be carried out in one of the following general ways:

1. **Conduct the reduction in place, i.e., the tube of Q-5 remains in place during reduction. The regeneration gas source is connected to flow through the Q-5 tube.**

2. **Remove the Q-5 tube from its normal position and place it in a simple regeneration apparatus. After regeneration securely seal off the Q-5 tube and place back in line.**

3. **If the Q-5 tube cannot be used in the regeneration apparatus (different fittings, etc.), transfer the Q-5 Reagent to a suitable regeneration container. After regeneration, transfer in an oxygen-free atmosphere the Q-5 back to the tube. Carefully replace the tube in its operating position avoiding oxygen contamination.**

The regeneration procedure is as follows:

1. **Heat the Q-5 Reagent to 200°C. This can best be accomplished by passage of heated inert gas (nitrogen, argon, etc.) through the container. A heating element on the Q-5 container can also be used to maintain the temperature.**

2. **Use a regeneration gas of 4-6 volume percent hydrogen remainder inert (helium, nitrogen, argon, carbon dioxide, methane). This gas can be purchased pre-mixed or the hydrogen and inert gases can be mixed before. Hydrogen concentrations in excess of six percent should not be used, as the high temperature generated will deactivate the Q-5.**

3. **Feed the gas at about 200°C at a rate of 500 volumes of total gas (STP)/hour/volume of Q-5. Should the bed temperature exceed 250°C, the hydrogen concentration should be reduced or inert flow increased and/or the gas temperature decreased. For larger beds of Q-5 Reagent (more than 10 pounds), at least one thermocouple should be installed in the bed to allow observation of bed temperature. For smaller beds, the temperature on reduction will not exceed 250°C when the above procedure is followed. (Note: A 2000 g bed of Q-5 Reagent, bed diameter 5”, bed height 6”, will give about 25°C temperature rise.)**

4. Continue the reduction for one hour.

5. Cool and purge the container with oxygen-free inert gas.

6. Impress a positive pressure of inert gas on the Q-5 Reagent tube.

The regeneration of Q-5 with hydrogen produces water. The fact that the exit regeneration gas contains water vapor can lead to condensation in cold exit lines and should be taken into consideration when designing the system. The initial reduction procedure is identical to the regeneration procedure outlined above.

After regeneration the utmost care should be exercised to assure that Q-5 Reagent is not exposed to oxygen. All lines to and from the Q-5 tube should be thoroughly purged with inert gas before putting the unit on stream.

USING ENGELHARD Q-5

After reduction, the Engelhard Q-5 Reagent will remove oxygen from various gases by simple passage through the reaction container. The Q-5 is effective over a wide temperature range, -40 to 200°C. The operating temperature selected usually depends on the temperature of the gas to be treated. Close control of the temperature is not necessary. Since the oxygen levels normally encountered in Q-5 treatment are quite low, there will be very little temperature rise in the Q-5 chamber.

The time between regenerations is determined in sizing of the Q-5 container (see "DESIGN OF ENGELHARD Q-5 UNITS"). Thus, from a knowledge of the oxygen content of the gas and the on-stream time of the unit, the user will know when it is necessary to regenerate.

The hardware requirements for a Q-5 unit are extremely simple. Any non-oxygen permeable material (metal, glass) is suitable for the Q-5 container. All fittings must be installed such that infusion of air is minimized. It is also important to assure that air leakage downstream from the unit is prevented. If the same container is to be used for regeneration, the temperature limitations of the material must be considered.

In both the regeneration and on-stream use of Q-5, extreme care should be taken to purge all lines to and from the container. In general it is advisable to remove Q-5 from its reaction container in its oxidized form, although no safety hazard exists. The Engelhard Q-5 Reagent merely warms up somewhat on exposure to air.